```
=> d his

     (FILE 'HOME' ENTERED AT 13:52:18 ON 17 FEB 2009)

     FILE 'CAPLUS' ENTERED AT 13:52:35 ON 17 FEB 2009
L1            13 S (PHTHALHYDRAZIDE OR PHTHALIMIDE) AND PENTADIENE

=> d 1-13 bib abs
```

L1 ANSWER 1 OF 13 CAPLUS COPYRIGHT 2009 ACS on STN
AN 2007:646507 CAPLUS
DN 147:249819
TI Development of Reliable Aqueous Solubility Models and Their Application in Druglike Analysis
AU Wang, Junmei; Krudy, George; Hou, Tingjun; Zhang, Wei; Holland, George; Xu, Xiaojie
CS Encysive Pharmaceuticals Inc., Houston, TX, 77030, USA
SO Journal of Chemical Information and Modeling (2007), 47(4), 1395-1404
CODEN: JCISD8; ISSN: 1549-9596
PB American Chemical Society
DT Journal
LA English
AB In this work, two reliable aqueous solubility models, ASMS (aqueous solubility based on mol. surface) and ASMS-LOGP (aqueous solubility based on mol. surface using calculated log P (ClogP) as a descriptor), were constructed by using atom type classified solvent accessible surface areas and several mol. descriptors for a diverse data set of 1708 mols. For ASMS (without using ClogP as a descriptor), the leave-one-out q2 and root-mean-square error (RMSE) were 0.872 and 0.748 log unit, resp. ASMS-LOGP was slightly better than ASMS (q2 = 0.886, RMSE = 0.705). Both models were extensively validated by three cross-validation tests and encouraging predictability was achieved. High throughput aqueous solubility prediction was conducted for a number of data sets extracted from several widely used databases. The authors found that real drugs are about 20-fold more soluble than the so-called druglike mols. in the ZINC database, which have no violation of Lipinski's "Rule of 5" at all. Specifically, oral drugs are about 16-fold more soluble, while injection drugs are 50-60-fold more soluble If the criterion of a mol. to be soluble is set to -5 log unit, about 85% of real drugs are predicted as soluble; in contrast only 50% of druglike mols. in ZINC are soluble The authors concluded that the two models could be served as a rule in druglike anal. and an efficient filter in prioritizing compound libraries prior to high throughput screenings (HTS).
RE.CNT 21 THERE ARE 21 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L1 ANSWER 2 OF 13 CAPLUS COPYRIGHT 2009 ACS on STN
AN 2006:829544 CAPLUS
DN 145:418716
TI meta-Directing cobalt-catalyzed Diels-Alder reactions
AU Hilt, Gerhard; Janikowski, Judith; Hess, Wilfried
CS Fachbereich Chemie, Philipps-Universitaet Marburg, Marburg, 35043, Germany
SO Angewandte Chemie, International Edition (2006), 45(31), 5204-5206
CODEN: ACIEF5; ISSN: 1433-7851
PB Wiley-VCH Verlag GmbH & Co. KGaA
DT Journal
LA English
OS CASREACT 145:418716
AB The regioselectivity of Diels-Alder reactions with neutral electron demand between 1,3-dienes with alkynes can be controlled by simple cobalt diimine complexes so that the meta-substituted cycloadducts are generated in good yields and excellent regioselectivity.
RE.CNT 27 THERE ARE 27 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L1 ANSWER 3 OF 13 CAPLUS COPYRIGHT 2009 ACS on STN
AN 2005:325744 CAPLUS
DN 142:397734
TI Preparation of prodrugs containing chemiluminescent and photochromic moieties for selective drug delivery
IN Mills, Randell L.; Wu, Guo-Zhang
PA USA
SO U.S. Pat. Appl. Publ., 199 pp.
CODEN: USXXCO
DT Patent
LA English
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 20050080260	A1	20050414	US 2004-828558	20040421
PRAI	US 2003-464354P	P	20030422		

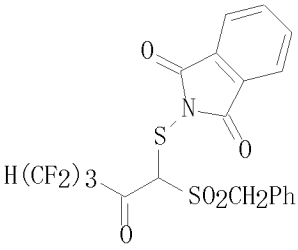
GI

* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

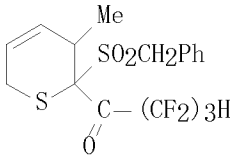
AB The invention relates to a method of synthesis of a chemical compound (I) having the formula A-B-C that may serve for applications such as drug delivery, where A is a chemiluminescent, moiety, B is a photochromic moiety, and C is a biol. active moiety where A-B-C may serve as a prodrug. Novel synthetic methods of the present invention to form the prodrug comprised the steps of (1) forming a benzophenone, (2) forming a diaryl ethylene, (3) attaching a phthalimide moiety to at least one of the aryl groups of the ethylene to form a phthalimide-ethylene conjugate, (4) condensing two ethylene-phthalimide conjugates to form a phthalimide-pentadiene conjugate, (5) converting the phthalimide to the phthalhydrazide by reaction with hydrazine to form a carrier compound according to the present invention, and (6) reacting the carrier compound with an nucleophilic moiety of the drug to form the corresponding prodrug. Alternatively the carrier can be prepared by using the halo-substituted diaryl ethylene to make the corresponding cationic leuco dye-like compound with known methods. The cationic compound then is protected by reacting with a nucleophile and coupled with the aminophathalimide by palladium-catalyzed amination to form the protected phthalimide-pentadiene conjugate. The latter is refluxed with hydrazine to convert its phthalimide to the phthalhydrazide and acidified to give the carrier. An addnl. aspect of the present invention relates to the use of these compds. as antiviral agents for the treatment of viral infections such as HIV and as anticancer agents for the treatment of cancers such as bowel, lung, and breast cancer.

L1 ANSWER 4 OF 13 CAPLUS COPYRIGHT 2009 ACS on STN
AN 2002:881452 CAPLUS
DN 140:181474
TI Product subclass 2: palladium-allyl complexes.
AU Friesen, R. W.
CS Merck Frosst Centre for Therapeutic Research, Kirkland, PE, H9H 3L1, Can.
SO Science of Synthesis (2002), 1, 113-264
CODEN: SSCYJ9
PB Georg Thieme Verlag
DT Journal; General Review
LA English
AB A review on preparation and application of palladium-allyl complexes.
RE.CNT 579 THERE ARE 579 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

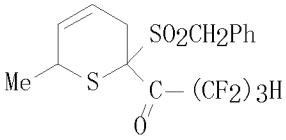
L1 ANSWER 5 OF 13 CAPLUS COPYRIGHT 2009 ACS on STN
AN 2002:438219 CAPLUS
DN 138:14032
TI Synthesis of 5,6-dihydro-2H-thiins and 2,3-dihydro-1,4-oxathiins based on 1-benzylsulfonyl-1,1-dihydropolyfluoroalkan-2-ones
AU Yemets, S. V.; Bandera, Yu. P.; Timoshenko, V. M.; Shermolvich, Yu. G.
CS Institute of Organic Chemistry, National Academy of Sciences of Ukraine, Kiev, 02094 -94, Ukraine
SO Journal of Fluorine Chemistry (2002), 115(2), 175-181
CODEN: JFLCAR; ISSN: 0022-1139
PB Elsevier Science B.V.
DT Journal
LA English
OS CASREACT 138:14032
GI



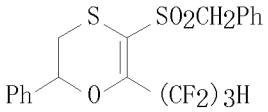
I



II



III



IV

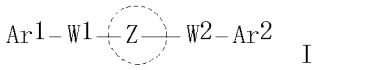
AB (Benzylsulfonyl)phthalimidothiopolyfluoroalkanones, e.g. I, were prepared from (benzylsulfonyl)polyfluoroalkanones, e.g. H(CF2)3COCH2SO2CH2Ph, and phthalimidosulfenyl chloride. Decomposition of I with evolution of phthalimide followed by Diels-Alder cycloaddn. with electron-rich 1,3-dienes, e.g. 1-methyl-1,3-butadiene, gave thiopyrans, e.g. II and III. Analogous Diels-Alder reaction of I with olefins, e.g. styrene, gave 1,4-oxathiins, e.g. IV, in yields of 64-85%.

RE.CNT 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

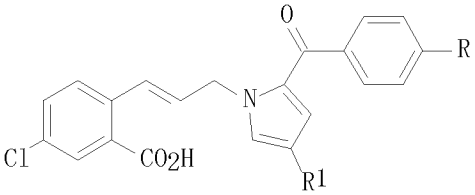
L1 ANSWER 6 OF 13 CAPLUS COPYRIGHT 2009 ACS on STN
AN 2002:107312 CAPLUS
DN 136:167389
TI Preparation of pyrrole, indole, thiophene, pyrazole, imidazole, and isothiazole derivatives as inhibitors of transforming growth factor-beta (TGF-β)
IN Tokunaga, Teruhisa; Hume, William Ewan; Kitoh, Makoto; Nagata, Ryu
PA Sumitomo Pharmaceuticals Co., Ltd., Japan
SO PCT Int. Appl., 215 pp.
CODEN: PIXXD2
DT Patent
LA Japanese
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2002010131	A1	20020207	WO 2001-JP6495	20010727
	W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW				
	RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
	AU 2001075794	A	20020213	AU 2001-75794	20010727
	CA 2416946	A1	20030122	CA 2001-2416946	20010727
	EP 1310485	A1	20030514	EP 2001-953325	20010727
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
	US 20030181496	A1	20030925	US 2003-352067	20030128
	US 6759429	B2	20040706		
	US 20040209939	A1	20041021	US 2004-840746	20040507
PRAI	JP 2000-229423	A	20000728		
	WO 2001-JP6495	W	20010727		
	US 2003-352067	A3	20030128		

OS MARPAT 136:167389
GI



I



III

AB The title compds. represented by the following formula (I) or pharmaceutically acceptable salts of these [wherein ring Z represents an optionally substituted pyrrole, indole, thiophene, pyrazole, benzene, imidazole, or isothiazole; W2 represents CO, SO2, CONR (R = H, alkyl), optionally substituted C1-4 alkylene or C2-4 alkenylene; Ar2 represents optionally substituted aryl or heteroaryl; and W1 and Ar1 mean the following: (1) W1 represents optionally substituted C1-4 alkylene or C2-4 alkenylene, Ar1 represents bicyclic heteroaryl having one to four N atoms or (2) W1 represents optionally substituted C2-5 alkylene, C2-5 alkenylene, C2-5 alkynylene, or -Y-W3 (wherein Y = O or cycloalkanediyl; W3 = optionally substituted C1-5 alkylene, C2-5 alkenylene, or C2-5 alkynylene), Ar represents optionally substituted aryl or monocyclic

heteroaryl substituted at ortho or meta position by CO2H, alkoxycarbonyl, optionally alkyl-substituted carbamoyl, cyclic aminocarbonyl, alkylsulfonylcarbonyl, arylsulfonylcarbonyl, alkylsulfonyl, etc.] or prodrugs or pharmacol. acceptable salts thereof are prepared These compds. are useful as fibroid inhibitors for organs or tissues. Thus, bromination of 3-(4-chloro-2-methoxycarbonylphenyl)-2-propenol (preparation given) by N-bromosuccinimide and PPh3 in CH2Cl2 at 0° for 10 min gave 3-(4-chloro-2-methoxycarbonylphenyl)-2-propenyl bromide (II). A THF solution of 2-(4-methylbenzoyl)pyrrole was added dropwise to a suspension of NaH in THF and the resulting solution was slowly added dropwise to a THF solution of II at 55° and stirred for 2 h to give 2-[3-[2-(4-methylbenzoyl)-1-pyrrolyl]-1-propen-1-yl]-5-chlorobenzoic acid Me ester which was saponified with aqueous NaOH in methanol and acidified with aqueous HCl to give III (R = Me, R1 = H). In a kidney fibroid model using a rat Thy-1 nephritis model, administration of III.Na (R = Me, R1 = H) at 15 mg/kg and Thy-1 (one of surface antigens of thymocyte) to rats lowered the level of hydroxyproline (fibroid index) in kidney compared to the control group administered only with Thy-1. III.Na (R = 2-morpholinoethoxy, R1 = Me) at 3 μM in vitro inhibited the TGF-β-induced production of proteoglycan in MRK-49F rat fibroblast cells by 99%.

RE.CNT 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L1 ANSWER 7 OF 13 CAPLUS COPYRIGHT 2009 ACS on STN
AN 2000:612647 CAPLUS
DN 133:178649
TI Conjugated diene rubber polymer for tire treads
IN Kim, Sam-Min; Bae, Jong-Pil; Yun, Dong-Il
PA Kumho Petrochemical Co., Ltd., S. Korea
SO Repub. Korea, No pp. given
CODEN: KRXXFC
DT Patent
LA Korean
FAN.CNT 1

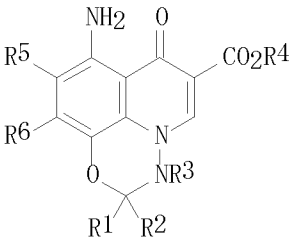
	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	KR 9510226	B1	19950912	KR 1992-21444	19921114
PRAI	KR 1992-21444		19921114		

AB The diene rubber polymer R-R' comprises 95-70 parts R component with structure comprising one of the conjugated diene rubber polymer selected from polybutadiene, styrene-butadiene copolymer, polyisoprene, styrene-isoprene copolymer, acrylonitrile-butadiene copolymer, polypentadiene, or butadiene-propene copolymer; 5-30 parts of R' component with structure comprising N-halophthalimide or N-haloalkyl phthalimide active group.

L1 ANSWER 8 OF 13 CAPLUS COPYRIGHT 2009 ACS on STN
AN 1995:997844 CAPLUS
DN 124:176157
OREF 124:32675a,32678a
TI Preparation of 8-amino-10-(azabicycloalkyl)pyrido[1,2,3-d,e][1,3,4]benzoxadiazines as antibacterial agents
IN Jaetsch, Thomas; Mielke, Burkhard; Petersen, Uwe; Schenke, Thomas; Bremm, Klaus-Dieter; Endermann, Rainer; Metzger, Karl-Georg; Scheer, Martin; Stegemann, Michael; Wetzstein, Heinz-Georg
PA Bayer A.-G., Germany
SO Eur. Pat. Appl., 70 pp.
CODEN: EPXXDW
DT Patent
LA German
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 682030	A1	19951115	EP 1995-106400	19950428
	EP 682030	B1	20000705		
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LI, NL, PT, SE				
	DE 4416622	A1	19951116	DE 1994-4416622	19940511
	AU 9516336	A	19951116	AU 1995-16336	19950407
	AU 689212	B2	19980326		
	TW 455589	B	20010921	TW 1995-84103734	19950417
	AT 194351	T	20000715	AT 1995-106400	19950428
	ES 2148372	T3	20001016	ES 1995-106400	19950428
	PT 682030	T	20001229	PT 1995-106400	19950428
	US 5679675	A	19971021	US 1995-434806	19950504
	CA 2148866	A1	19951112	CA 1995-2148866	19950508
	IL 113650	A	20000217	IL 1995-113650	19950508
	CN 1113243	A	19951213	CN 1995-105716	19950510
	CN 1042132	C	19990217		
	ZA 9503776	A	19960116	ZA 1995-3776	19950510
	HU 71611	A2	19960129	HU 1995-1377	19950510
	HU 219301	B	20010328		
	JP 08073468	A	19960319	JP 1995-136119	19950510
	RU 2138504	C1	19990927	RU 1995-107150	19950510
	HU 219562	B	20010528	HU 2000-337	19950510
	GR 3034280	T3	20001229	GR 2000-401967	20000830
PRAI	DE 1994-4416622	A	19940511		
	HU 1995-1377	A	19950510		

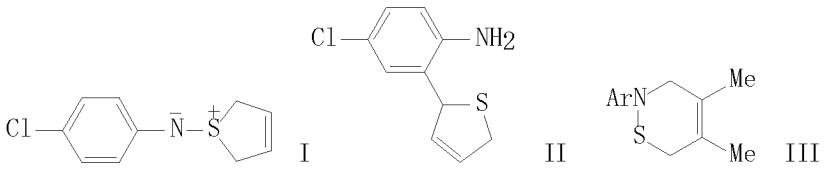
OS MARPAT 124:176157
GI



I

AB Title compds. [I; R1 = H, (halo)alkyl, hydroxyalkyl; R2 = H or Me; R3 = H or alkyl; R4 = H, (un)substituted alkyl, 5-methyl-2-oxo-1,3-dioxol-4-ylmethyl; R5 = H or halo; R6 = (un)substituted 8-azabicyclonon-2- or -3-en-8-yl, 2,8-diazabicyclononan-8-yl, etc.] were prepared Thus, I (R1 = R2 = R4 = H, R3 = Me, R5 = F)(II; R6 = F) was condensed with 2-oxa-5,8-diazabicyclo[4.3.0]nonane to give II [R6 = 2-oxa-5,8-diazabicyclo[4.3.0]nonan-8-yl] which had MIC of ≤0.015 and 0.125 (units not given) against Escherichia coli ATCC 25922 and Staphylococcus aureus ICB 25701, resp.

L1 ANSWER 9 OF 13 CAPLUS COPYRIGHT 2009 ACS on STN
AN 1994:578815 CAPLUS
DN 121:178815
OREF 121:32467a,32470a
TI Diels-Alder and ene reactions of new transient thionitrosoarenes (Ar-N=S) and thionitrosoheteroarenes (Het-N=S) generated from N-(arylaminosulfanyl)- and N-(heteroarylaminosulfanyl)phthalimides : synthesis of cyclic and acyclic sulfenamides
AU Bryce, Martin R.; Heaton, Julie N.; Taylor, Paul C.; Anderson, Martin
CS Dep. Chem., Univ. Durham, Durham, DH1 3LE, UK
SO Journal of the Chemical Society, Perkin Transactions 1: Organic and Bio-Organic Chemistry (1972-1999) (1994), (14), 1935-44
CODEN: JCPRB4; ISSN: 0300-922X
DT Journal
LA English
OS CASREACT 121:178815
GI

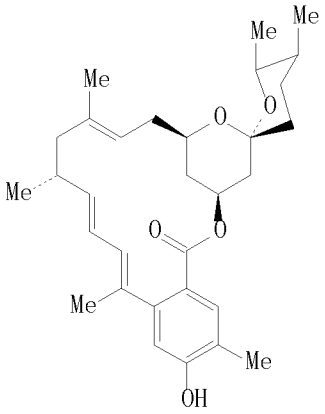


AB A series of new N-(arylaminosulfanyl)- and N-(heteroarylaminosulfanyl) phthalimides (3) has been prepared by reaction of chlorosulfanylphthalimide with the trimethylsilyl derivative of the appropriate arylamine or heteroarylamine. On treatment with triethylamine at room temperature, most of these compds. 3 fragment to yield transient thionitroso species, Ar-N=S and Het-N=S, which have been intercepted, generally in good yield, with conjugated dienes (2,3-dimethylbuta-1,3-diene, isoprene, chloroprene and penta-1,3-diene) to yield cyclic 1,2-thiazine Diels-Alder adducts and with alkenes (1-methylcyclohexene, α-pinene and β-pinene) to yield acyclic ene adducts. Competitive Diels-Alder and ene addition is observed with dimethylbutadiene and isoprene. The regiochem. of addition of unsym. dienes to thionitroso species has been elucidated. Sulfilimine I rearranges quant. to the dihydrothiophene derivative II, thereby excluding sulfilimines as intermediates in the formation of 1,2-thiazine adducts III.

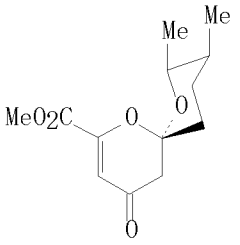
L1 ANSWER 10 OF 13 CAPLUS COPYRIGHT 2009 ACS on STN
AN 1990:95635 CAPLUS
DN 112:95635
OREF 112:16199a,16202a
TI Comparative effects of heterocyclic compounds on inhibition of lettuce fruit germination
AU Reynolds, T.
CS Jodrell Lab., R. Bot. Gardens, Kew/Richmond/Surrey, UK
SO Journal of Experimental Botany (1989), 40(212), 391-404
CODEN: JEBOA6; ISSN: 0022-0957
DT Journal
LA English
AB The mols. of many biol. active plant constituents contain heterocyclic ring systems. Inhibitory effects of a number of heterocyclic compds. and their alicyclic and open-chain analogs on lettuce (Lactuca sativa cv. Great Lakes) germination were therefore determined under specific conditions. The most obvious property which correlates chemical structure with biol. activity was lipophilicity. However, other less obvious factors play a part. The inhibitory activity of coumarin, for instance, was much greater than would be expected in comparison with compds. of related structures. In general, substitution of a C atom in a ring structure by O or N has either little effect or a lowering effect on activity, unless the increased solubility in water allows an inhibitory concentration to be reached which did not occur with the carbocyclic compound However, introduction of unsatn. increases activity markedly, especially with some of the indole compds.

L1 ANSWER 11 OF 13 CAPLUS COPYRIGHT 2009 ACS on STN
AN 1987:72791 CAPLUS
DN 106:72791
OREF 106:11893a,11896a
TI A method for calculation of the aqueous solubility of organic compounds by using new fragment solubility constants
AU Wakita, Keiko; Yoshimoto, Masafumi; Miyamoto, Shuichi; Watanabe, Hidetoshi
CS Chem. Res. Lab., Sankyo Co. Ltd., Tokyo, 140, Japan
SO Chemical & Pharmaceutical Bulletin (1986), 34(11), 4663-81
CODEN: CPBTAL; ISSN: 0009-2363
DT Journal
LA English
AB For the calcn. of the aqueous solubility of organic compds., new fragment solubility consts. (fs) were defined and empirically determined on the basis of compiled data from the literature. First, 6 fundamental fs values were determined from data on 46 liquid aliphatic hydrocarbons. These fs values were fixed, and data on 249 liquid aliphatic compds. with diverse functional groups were employed to optimize another 19 fs values of the groups. Then, 15 fs values of aromatic compds. were calculated based on the solubility data on 58 aromatic liqs. and the aliph fs values. There is a linear relation between the logarithms of the aqueous solubilities of organic liqs. and the octanol-water partition consts. (log P), and the water solubilities can be calculated by using the correlation equation and log P values. Thus, a method to calculate the aqueous solubilities of organic liqs. simply, directly and more accurately on the basis of fs was proposed. Furthermore, the calcn. of the water solubilities of organic solids was attempted with a correction based on the m.ps., in addition to using the fs values.

L1 ANSWER 12 OF 13 CAPLUS COPYRIGHT 2009 ACS on STN
AN 1987:49831 CAPLUS
DN 106:49831
OREF 106:8247a,8250a
TI Total synthesis of (+)-milbemycin β3
AU Barrett, Anthony G. M.; Carr, Robin A. E.; Attwood, Stephen V.; Richardson, Geoffrey; Walshe, Nigel D. A.
CS Dep. Chem., Northwestern Univ., Evanston, IL, 60201, USA
SO Journal of Organic Chemistry (1986), 51(25), 4840-56
CODEN: JOCEAH; ISSN: 0022-3263
DT Journal
LA English
OS CASREACT 106:49831
GI



I



II

AB In the total synthesis of (+)-milbemycin β3 (I) the key features are the preparation of I from only 2 chiral pool starting materials (S)-(+)-citronellene and (S)-(-)-propylene oxide. The spiro ketal moiety II was constructed using the condensation reaction of 5(S),6(R)-dimethyltetrahydro-2-pyranone with 2,4-dilithioxy-1,1,1-trimethoxy-2,4-pentadiene. The macrolide was constructed using Julia-Lythgoe and benzylic anion chemical Mitsunobu closure of the lactone ring was highly efficient. The synthesis is concise and with the exception of the construction of Δ14 is highly stereochem. controlled.

L1 ANSWER 13 OF 13 CAPLUS COPYRIGHT 2009 ACS on STN
AN 1966:43235 CAPLUS
DN 64:43235
OREF 64:8015g-h,8016a-h
TI Highly chlorinated aliphatic amines and their basicity
AU Roedig, Alfred; Grohe, Klaus; Maerkl, Gottfried
CS Chem. Inst. Univ. Wuerzburg, Germany
SO Chemische Berichte (1966), 99(1), 121-9
CODEN: CHBEAM; ISSN: 0009-2940
DT Journal
LA German
AB Primary aliphatic amines of the type RCH2NH2, wherein R is a highly chlorinated, saturated or unsatd. aliphatic group, were prepared by LiAlH4 reduction of the corresponding nitriles or carboxamides as well as by the Gabriel synthesis. Their basic dissociation consts. were determined potentiometrically and compared with each other and with those of non-halogenated and fluorinated amines. CCl2: CClCONH2 (I) (908 g.) and 382 g. P2O5 heated slowly in vacuo to 190-200° yielded 750 g. CCl2: CClCN (II), b11 38-40° ,m. 18-20° ,n20.5D 1.5100. C2Cl5COCl (238 g.) in 100 cc. Et2O added dropwise with stirring to 200 cc. cold concentrated NH4OH gave 212 g. (crude) C2Cl5CONH2 (III), m. 245° (30% EtOH-ligroine, b. 90-100°). III (305 g.) and 207 g. POCl3 heated 1 hr. at 100-15° ,treated dropwise with 15 cc. C5H5N, and refluxed 3-4 hrs. yielded 260 g. C2Cl5CN (IV), m. 150.5-1.5° (aqueous MeOH). II (30 g.) treated under irradiation with a 500-w. lamp at 150-60° with dry Cl yielded 29 g. IV. CCl2:CClCCl:CClCONH2 (236 g.) and 102 g. POCl3 heated 3-4 hrs. at 100-10° and poured onto ice gave 200 g. CCl2: CClCCl:CClCN, m. 46° (petroleum ether), b2-3 76-8° CHCl2CN (22 g.) in 60 cc. Et2O added dropwise with stirring during 2-3 hrs. at -20° to 7.8 g. LiAlH4 in 250 cc. dry Et2O, stirred 15 min., and treated with 48 cc. saturated aqueous NaCl gave 1.2 g.CHCl2CH2NH2 (V), b58 60-4° . V in dry Et2O treated with dry HCl, and the product sublimed at 150-60° /11mm. gave V.HCl, m. 158-62° (sealed capillary) (absolute EtOH). V with PhNCO in dry C6H6 gave CHCl2CH2NHCONHPh, m. 135-6° (2:1 MeOH H2O or EtOH). CCl3CN (64.8 g.) in 100 cc. Et2O added dropwise with stirring and cooling during 2-3 hrs. to 13.8 g. LiAlH4 in 700 cc. dry Et2O, stirred 45 min., and decomposed with 110 cc. saturated aqueous NaCl gave 35 g. CCl3CH2NH2, (VI), b20 43° ,n20D 1.4912;VI.HClm. 244-5° (decomposition) (absolute EtOH). VI with PhNCO gave CCl3CH2NHCONHPh, m. 165-5.5° (75% MeOH). VI with BzCl and alkali gave CCl3CH2NHBz, m. 137-8° (ligroine, b. 90-110°). VI with CCl3COCl and alkali yielded CCl3CH2NHCOCCl3, m. 132.5-3.5° (ligroine). VI with CCl2:CClCCl:CClCOCl and aqueous alkali gave CCl3CH2NHCOCCl:CClCCl: CCl2, m. 80-2° (petroleum ether). CH2ClCCl2CN (31.5 g.) in 50cc. dry Et2O stirred 2 hrs. with 7.6 g. LiAlH4 in 400 cc. Et2O gave 21 g. CH2Cl-CCl2CH2NH2 (VII), b12 68-9° ,n20D 1.5019. VII.HCl with 0.88 g. KOCN in a little H2O yielded (CH2ClCCl2CH2NH)2CO, m. 95-5.5° (CHCl3). VII with PhNCO in dry C6H6 gave CH2ClCCl2-CH2NHCONHPh, m. 112° (1:1 MeOH-H2O). III (49 g.) in 250 cc. dry Et2O added with cooling and stirring during 1.5 hrs. to 15.2 g. LiAlH4 in 260 cc. Et2O, stirred 1 hr. at room temperature, and refluxed 9 hrs. yielded 14 g. yellow CCl3CCl2CH2NH2 (VIII),b0.25-0.3 26-8° n20D 1.5210. IV (45.5 g.) in 100 cc. dry Et2O treated with cooling and stirring with 7.6 g. LiAlH4 in 400 cc. Et2O during 2 hrs. and stirred 45 min. at 0° gave 19 g. VIII, b0.2, 25-7° ;VIII.HCl decompose 226-9° (sealed capillary) (sublimed at 0.5 mm.) (absolute EtOH); N-Bz derivative m. 182-3° (2:1 MeOH-H2O and ligroine, b. 130-80°). VIII with CCl2:CClCOCl and aqueous alkali gave CCl2:CClCONHCH2CCl2CCl3, m. 132.5-3.5° (1:1 MeOH-H2O and ligroine, b. 90-110°). CCl2: CClCH2OH: (30 g.) and 50.5 g. PBr3 heated 0.5 hr. at 185° yielded 32 g. lacrimatory CCl2:CClCH2Br (IX), b11 67-8° n20D 1.5560. IX (4.45 g.) in 25 cc. HCONMe2 and 4.1 g. K phthalimide heated briefly on a water bath gave 5.7 g. crude 1,1,2-trichloro-3-phthalimido-1-propene (X), m. 114.5-15.5° (MeOH and ligroine). X (68 g.) in 460 cc. MeOH refluxed 1 hr. with 12.5 g. 94% N2H4.H2O, diluted with 250 cc. H2O, concentrated, and refluxed 1 hr. with 300 cc. concentrated HCl gave 21 g. CCl2:CClCH2NH2 (XI),

b11, 63-4° ;XI.HCl decomposed 204-8° (sealed capillary)
(sublimed at 0.01 mm.) (absolute EtOH). I (35 g.) in 200 cc. Et2O reduced
with 7.9 g. LiAlH4, and the crude product treated in Et2O with dry HCl
yielded 12 g. crude XI.HCl. II (31.7 g.) in 100 cc. Et2O added dropwise
during 2-3 hrs. at 0° to 8.5 g. LiAlH4 in 350 cc. dry Et2O and
stirred 0.5 hr. at 0° ,and the crude product treated in Et2O with
dry HCl yielded 19 g. XI.HCl. XI.HCl (1.2 g.) and 0.8 g. KOCN gave
(CCl2:CClCH2NH)2CO, m. 146-7.5° (H2O). XI was converted to
CCl2:CClCH2NHCONHPh, m. 161.5-2.5° (MeOH), and to the N-Bz derivative,
m. 124-5° (CCl4 or ligroine). CCl2:CClCCl:CClCH2Br (51.9 g.) in
160 cc. HCONMe2 treated with stirring with 30.5 g. K phthalimide
in portions and heated 10 min. on a water bath yielded 28 g. crude
1,1,2,3,4-pentachloro-5-phthalimido-1,3-pentadiene (XII), m.
130.5-1.5° (petroleum ether and AcOEt). XII (17.1 g.) and 3.4 g.
94% N2H4.H2O in 180 cc. MeOH refluxed 1 hr., diluted with 75 cc. H2O,
concentrated, and refluxed 1 hr. with 70 cc. concentrated HCl gave 4.5 g. CCl2:
CClCCl: CClCH2NH2 (XIII), b0.08 66-8° ,n20D 1.5660; XIII.HCl m.
207-9° (sealed capillary) (sublimed at 0.2 mm.) (absolute EtOH). XIII
with BzCl and aqueous alkali gave the N-Bz derivative, m. 173-4° (ligroine,
b. 130-80° ,and 90% EtOH). XIII with PhNCO in dry C6H6 yielded
CCl2:CClCCl:CClCH2NHCONHPh, m. 174-5° (80% EtOH). XI (20 g.) kept
3 weeks at room temperature, and the resulting black-brown resin extracted with H2O
gave from the ext 6.5 g. XI.HCl. VIII (12 g.) distilled at 84° /12mm.
gave about 1 g. CCl3CCl: CHNH2 which with PhNCO in C6H6 yielded
CCl3CCl:CHNHCONHPh, m. 137.5-8.5° (75% MeOH). The base consts. KB
were determined for the following compds.: CH2ClCH2NH2, 3.6 +
10-8/21° (7.31); V, 5.6 + 10-8/20° (9.78); VI, 1.8
+ 10-9/20° (11.71); CF3CH2NH2, 4.0 + 10-9/20° ;
VII, 1.0 + 10-8/23° ;XI, 1.9 + 10-7/22° ;XIII,
6.0 + 10-8/22° . The values in parentheses are the free
energies of protonation in kcal./mole.

```
=> d his full

     (FILE 'HOME' ENTERED AT 13:52:18 ON 17 FEB 2009)

     FILE 'CAPLUS' ENTERED AT 13:52:35 ON 17 FEB 2009
L1            13 SEA ABB=ON  PLU=ON  (PHTHALHYDRAZIDE OR PHTHALIMIDE) AND
                 PENTADIENE
                 D 1-13 BIB ABS


     FILE HOME

     FILE CAPLUS

     Copyright of the articles to which records in this database refer is
     held by the publishers listed in the PUBLISHER (PB) field (available
     for records published or updated in Chemical Abstracts after December
     26, 1996), unless otherwise indicated in the original publications.
     The CA Lexicon is the copyrighted intellectual property of the
     American Chemical Society and is provided to assist you in searching
     databases on STN.  Any dissemination, distribution, copying, or storing
     of this information, without the prior written consent of CAS, is
     strictly prohibited.

     FILE COVERS 1907 - 17 Feb 2009  VOL 150 ISS 8
     FILE LAST UPDATED: 16 Feb 2009  (20090216/ED)

     Caplus now includes complete International Patent Classification (IPC)
     reclassification data for the third quarter of 2008.

     CAS Information Use Policies apply and are available at:

     http://www.cas.org/legal/infopolicy.html

     This file contains CAS Registry Numbers for easy and accurate
     substance identification.

=> log h
COST IN U.S. DOLLARS                            SINCE FILE      TOTAL
                                                   ENTRY    SESSION
FULL ESTIMATED COST                                47.22      47.44

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)      SINCE FILE      TOTAL
                                                   ENTRY    SESSION
CA SUBSCRIBER PRICE                               -10.66     -10.66

 SESSION WILL BE HELD FOR 120 MINUTES
STN INTERNATIONAL SESSION SUSPENDED AT 13:54:15 ON 17 FEB 2009
```